1,158,591

PATENT SPECIFICATION

NO DRAWINGS

1,158,591

Date of filing Complete Specification: 8 July, 1966.
Application Date: 14 July, 1965. No. 29945/65.
Complete Specification Published: 16 July, 1969.

Index at acceptance:—C3 C1C; C1 J(7, 12, 17, 26, 35); C3 B(1C1, 1C6X, 1D2C, 1G, 1N4H, 1N6A); C3 P(14A, 14C8A, 14C8B, 14C15, 14D1B, 14D2C, 14F2, 14K4, 14K7, 14K8, 14K11, 14P4C); C3 R(1A, 1C1, 1C6X, 1G, 1P1, 1P2, 1P4, 32C1, 32C6X, 32D1, 32D6C, 32G1); T(6F2, 6J3B, 6J3E)

Int. Cl.:—C 08 g 51/04

COMPLETE SPECIFICATION

Improvements in Thermal Insulation

ERRATUM

SPECIFICATION No. 1,158,591

Page 1, Index at acceptance:— *after* "32G1);" *insert* "C3"

THE PATENT OFFICE
12th August 1969

if they are in continuous contact with a flame, or are maintained at temperatures above 200° C, these organic materials decompose and their thermal insulating effect is thereby lost.

The object of the invention is the overcoming of these disadvantages.

According to the invention a method of forming thermal insulation material for use in a cavity or in a heat shield comprises admixing a cellular inorganic particulate material with a curable synthetic resin binder which is either itself flame-retardant or is rendered so by the addition of a flame-retardant agent thereto and curing the resin *in situ*, the amount of resin being sufficient to form the material into a coherent mass after such curing. A curable resin is, of course, cross-linking.

Preferably the synthetic resin is used in an amount of from 5% upwards by volume of

Preferred synthetic resin binders are:—

1. Liquid one-stage phenol-formaldehyde resins in which a flame-retardant agent is incorporated.

2. Liquid polyester resins of the unsaturated type, rendered flame-retardant by the use of hexa chloroendomethylene tetrahydrophthalic acid as part of the acid component of the polyester and which may be cured by means of styrene or methyl methacrylate monomer or diallyl phthalate or triallyl phosphate, with the appropriate peroxy catalyst.

3. Liquid epoxy resins with diamine or acid anhydride curing agent with a flame-resistant agent incorporated in the system.

4. Polyurethane resin systems based on polyethers or polyesters, with a polyisocyanate as the curing agent, and with a flame-resistant agent in the system.

5. Liquid silicone polymer, essentially poly-

[*Price 4-*

SEE ERRATA SLIP ATTACHED

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Int. Cl.:—C 08 g 51/04

COMPLETE SPECIFICATION

Improvements in Thermal Insulation

I, CYRIL AUBREY REDFARN, of Quality House, Chancery Lane, London, W.C.2, a British Subject, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to improvements in inorganic cellular materials for thermal insulation.

Cavity walls, for example, in houses and other building structures, in refrigerators, in ships holds and railway wagons are often filled with light-weight cellular organic materials, of a density of 1 to 2 pounds per cubic foot, such as polyurethane foam and expanded polystyrene. Such cellular materials can be rendered flame-resistant by the incorporation of flame-retarding compounds therein. However, whilst light-weight cellular organic materials that are flame-resistant can be produced, such materials are combustible in that, if they are in continuous contact with a flame, or are maintained at temperatures above 200° C, these organic materials decompose and their thermal insulating effect is thereby lost.

The object of the invention is the overcoming of these disadvantages.

According to the invention a method of forming thermal insulation material for use in a cavity or in a heat shield comprises admixing a cellular inorganic particulate material with a curable synthetic resin binder which is either itself flame-retardant or is rendered so by the addition of a flame-retardant agent thereto and curing the resin *in situ*, the amount of resin being sufficient to form the material into a coherent mass after such curing. A curable resin is, of course, cross-linking.

Preferably the synthetic resin is used in an amount of from 5% upwards by volume of the final admixture. The resin and particulate material may be simultaneously sprayed into the position which is finally to be occupied by the thermal insulation material, the two streams of material being so directed that the resin is uniformly distributed throughout the particulate material. In order to ensure that the final thermal insulation is more fully flame-retardant, it is preferred to incorporate a flame-retarding agent into the said synthetic resin, except in those cases in which the resin is itself sufficiently flame-retardant.

The invention also includes such thermal insulation material.

Suitable inorganic materials are pumice stone reduced to particles of about 1 mm. exfoliated vermiculite particles, expanded perlite particles bulking about 2 to 5 pounds per cubic foot, ceramic or earthenware microballoons of substantially unbroken skin formation and microballoons or hollow beads both made of metal.

Preferred synthetic resin binders are:—

1. Liquid one-stage phenol-formaldehyde resins in which a flame-retardant agent is incorporated.

2. Liquid polyester resins of the unsaturated type, rendered flame-retardant by the use of hexa chloroendomethylene tetrahydrophthalic acid as part of the acid component of the polyester and which may be cured by means of styrene or methyl methacrylate monomer or diallyl phthalate or triallyl phosphate, with the appropriate peroxy catalyst.

3. Liquid epoxy resins with diamine or acid anhydride curing agent with a flame-resistant agent incorporated in the system.

4. Polyurethane resin systems based on polyethers or polyesters, with a polyisocyanate as the curing agent, and with a flame-resistant agent in the system.

5. Liquid silicone polymer, essentially poly-

SEE ERRATA SLIP ATTACHED

dimethyl silicone, but with some reactive hydrogen on the molecules so that they will cure with a catalyst such as dibutyl tin dilaurate.

6. Semi-inorganic resins that are thermosettable polynuclear phenolic resins which have free phenolic groups with phospho-, boro-, phosphoboro- or phospho- nitrogen linkages between the phenolic nuclei and which may be cured with a formaldehyde donor such as hexamethylene tetramine or paraformaldehyde.

A number of flame-retarding agents for adding to synthetic resins and polymers, are readily available. These commonly are bromine containing organic compounds that are compatible with the particular resin used.

Bonding systems for cellular inorganic particles according to the invention to be used as thermal insulating materials in cavities and as layers on one or both sides of sheets of metal, asbestos board and other incombustible materials have several advantages. For instance, there is no substantial change in the apparent density of the bonded composition and no significant amount of volatile matter is given off as the system cures. Only the minimum amount of bonding material to give a coherent mass need be used, the compositions can be cold or heat cured and the final product is flame-resistant.

A useful bond can be obtained with as little as 5% by volume of bonding material but if improved physical strength is required as much as 35% by volume bonding materials in the composite mass is required (the percentages being before mixing), but in such latter products the apparent density will be higher than when using the lower percentages of the bonding material. (On mixing there is a volume shrinkage because some of the resin gets into the cellular filler through broken cell walls.)

In one embodiment of the invention, for filling a wall cavity, the cellular inorganic material, expanded perlite particles of about ½ to 1 mm diameter, is blown into the cavity in conjunction with an atomised spray of polyester resin of the type described. Both streams of blown particles are metered so that the resin component amounts to about 5% by volume of the composition and the streams are mixed so that the resin particles attach themselves to the cellular particles and form a bond between them.

Such a structure is flame-resistant and of a high degree of incombustibility because of the low resin bond content. When a higher degree of heat resistance is required a silicone polymer as described is used and for still better heat resistance, a semi-inorganic resin as described above is used.

The mixed blowing method can be used for applying coatings of resin-bonded inorganic cellular materials. When the composition is to be applied by spreading and trowelling methods the resin bond content usually needs to be somewhat higher and is about 1/3 of the volume of the total mix.

In a modification of the invention, the bonding resin systems have embodied in them foaming agents for the purpose of expanding the resin in the manner well-known in the art. By this means the increased density effect due to the resin bond is compensated, but the structure is physically weaker than that obtained with a non-expanding resin system.

WHAT I CLAIM IS:—

1. A method of forming thermal insulation material for use in a cavity or in a heat shield, comprising admixing a cellular inorganic particulate material with a curable synthetic resin binder which is either itself flame-retardant or is rendered so by the addition of a flame-retardant agent thereto and curing the resin *in situ*, the amount of resin being sufficient to form the material into a coherent mass after such curing.

2. A method according to Claim 1 in which the synthetic resin binder is used in an amount of from 5% upwards by volume of the admixture.

3. A method according to Claim 1 or Claim 2 in which the resin and the particulate material are simultaneously sprayed into the position which is finally to be occupied by the thermal insulation material, the two streams of material being so directed that the resin is uniformly distributed throughout the particulate material.

4. A method according to any of Claims 1, 2 or 3 in which the particulate material is pumice stone.

5. A method according to any of Claims 1, 2 or 3 in which the particulate material consists of particles of exfoliated vermiculite.

6. A method according to any of Claims 1, 2 or 3 in which the particulate material consists of expanded particles of perlite.

7. A method according to any of Claims 1, 2 or 3 in which the particulate material consists of ceramic or earthenware microballoons of substantially unbroken skin formation.

8. A method according to any of claims 1, 2 or 3 in which the particulate material consists of metal microballoons or metal hollow beads.

9. A method according to any of the preceding claims in which the synthetic resin binder is a flame-retardant phenolformaldehyde resin in which a flame-retardant agent is incorporated.

10. A method according to any of claims 1 to 8 in which the synthetic resin binder is a liquid polyester resin of the unsaturated type, rendered flame-retardant by the use of hexa chloro-endomethylene tetrahydrophthalic acid as part of the acid component of the

polyester and cured by means of styrene or methyl methacrylate monomer or diallyl phthalate or triallyl phosphate, with the appropriate peroxy catalyst.

11. A method according to any of claims 1 to 8 in which the synthetic resin binder is a liquid epoxy resin with diamine or acid anhydride curing agent with a flame-resistant agent incorporated in the system.

12. A method according to any of claims 1 to 8 in which the synthetic resin binder is a polyurethane resin system based on polyethers or polyesters, with a polyisocyanate as the curing agent, and with a flame-resistant agent in the system.

13. A method according to any of claims 1 to 8 in which the synthetic resin binder is a polydimethyl silicone having some reactive hydrogen on the molecules so that they will cure with a catalyst.

14. A method according to any of claims 1 to 8 in which the synthetic resin binder is a polynuclear phenolic resin having free phenolic groups with phospho-, boro-, phosphoboro- or phospho- nitrogen linkages between the phenolic nuclei and reacted with a formaldehyde donor.

15. A thermal insulation material comprising an admixture of a cellular inorganic particulate material as used in the method of any of claims 4 to 8 with a synthetic resin binder as used in the method of any of claims 9 to 14.

16. A method of forming thermal insulation material in a cavity or in a heat shield substantially as described.

17. A thermal insulation material for use in a cavity or in a heat shield substantially as described.

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